UNIVERSITY OF CALIFORNIA
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FUNDAMENTAL STUDIES RELATED TO THE ORIGIN AND NATURE OF CREEP OF METALS

Ninth Technical Report

Recent Observations on the Motion of Small Angle Dislocation Boundaries

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INTRODUCTION

The possibility that a small-angle crystal boundary might consist of an array of dislocations was pointed out by Burgers[1] and by Shockley and Read[2]. Experimental verification of this concept was obtained in some recent work[3,4]. It is the purpose of this paper to report further observations on the properties of small angle dislocation boundaries in zinc in the temperature range -196° to 400°C.

After annealing at a temperature close to the melting point, it is presumed that a small angle boundary consists of an approximately evenly-spaced array of edge dislocations. For a 1° boundary in zinc the spacing between dislocations is about 60 interplanar distances. When such an array has been caused to move by an applied shear stress, the volume of crystal through which the array has passed has undergone a shear strain. For a 1° boundary a shear strain of about 2 percent is produced; the magnitude of the strain is determined by the angle of the boundary. The motion of small angle boundaries can therefore be considered as a particularly uniform type of plastic deformation in which single dislocations traverse every 10th to 100th slip plane rather than 100 to 1,000 dislocations traversing much more widely separated planes. Continued displacement of the boundary involves an enlargement of the strained volume of crystal. As has been pointed out[5], the stress required to set an array of dislocations forming a boundary into motion is approximately the same as the yield stress for the material. Since motion of a boundary only involves movement of dislocations already existing, this implies that the stress necessary to move dislocations may determine the yield strength of zinc rather than the stress necessary to generate new dislocations.

EXPERIMENTAL TECHNIQUES

The method of preparing small angle boundary specimens has been described in an earlier paper[4]. Recently an improvement in the shape of specimens was made, in that 0.14" x 0.2" x 1.0" rectangular bars were employed in place of the earlier discs. The rectangular bar specimens were cut to size by means of an acid sawing technique described elsewhere[6]. A specimen of this shape offered the advantage of constant average shear stress on the boundary during its motion. A typical specimen containing a simple edge type boundary is shown in Fig. 1. The pronounced change in light intensity existing at the boundary is due to the relative change in orientation of the basal plane across the boundary. The boundary is also visible on the side or $(1\bar{1}00)$ face of the specimen. This effect was attributed to the reflection characteristics of a minute line grating produced by etching during the acid sawing operation. Etched valleys constituting the grating were visible at 500X and appeared randomly spaced but were aligned parallel to the basal plane. The tilting about the $[1\bar{1}00]$ axis was sufficient to cause this optical effect.

The specimens were loaded and observed both at low and high temperature in a manner shown photographically in Fig. 2 and schematically in Fig. 3. For high temperature studies, the specimen was supported in a small electrical resistance furnace. At liquid nitrogen temperatures the specimen was immersed in a Dewar flask. Both the furnace and vacuum flask contained viewing ports of optically flat glass to facilitate the microscopic observation of the boundaries and their motion. Boundary angles were measured optically with a long focal length goniograph capable of an angular resolution of approximately 30 seconds. (See Appendix for details)

FIG. 1 MACROPHOTOGRAPH SHOWING A 0.73° EDGE DISLOCATION BOUNDARY IN A ZINC CRYSTAL SPECIMEN OF THE TYPE USED FOR BOUNDARY MOTION STUDIES. 4X.

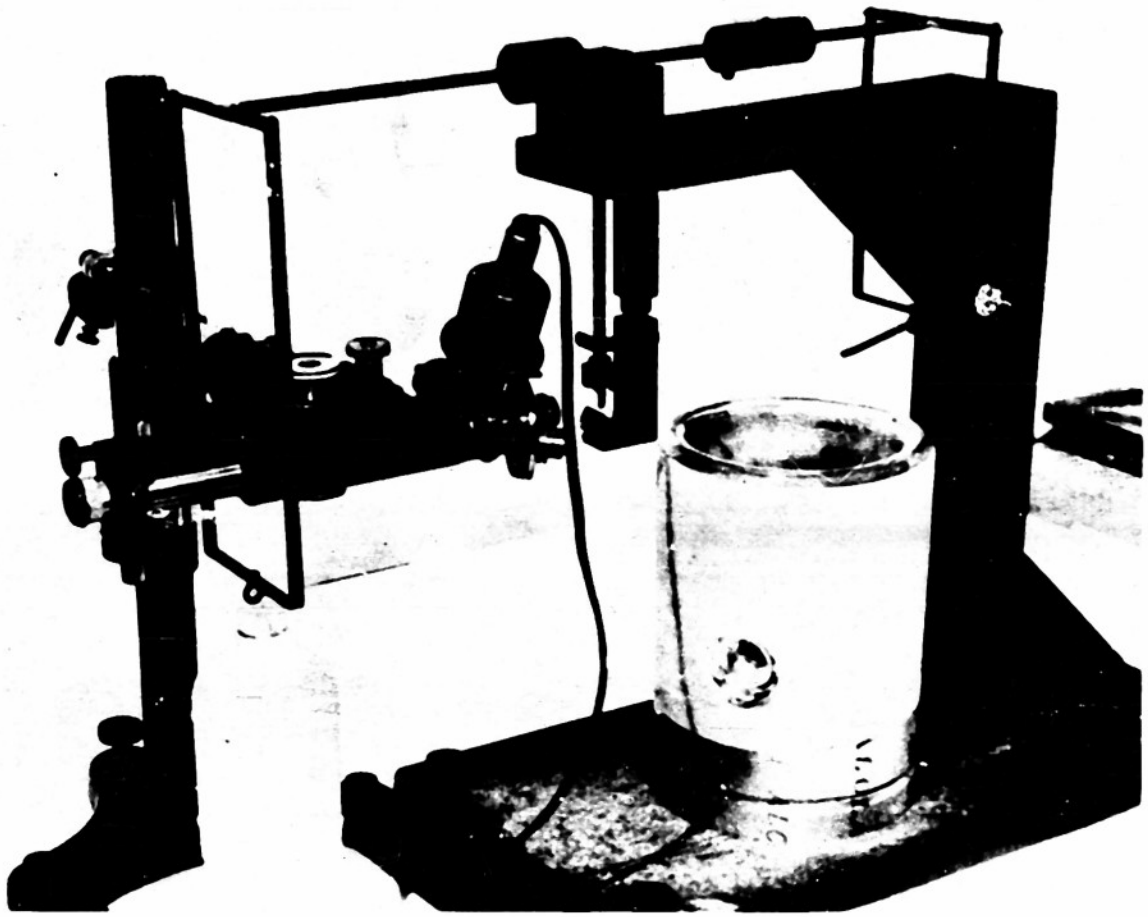


FIG. 2 EQUIPMENT USED IN STUDYING THE MOTION OF SMALL ANGLE BOUNDARIES IN ZINC CRYSTALS.

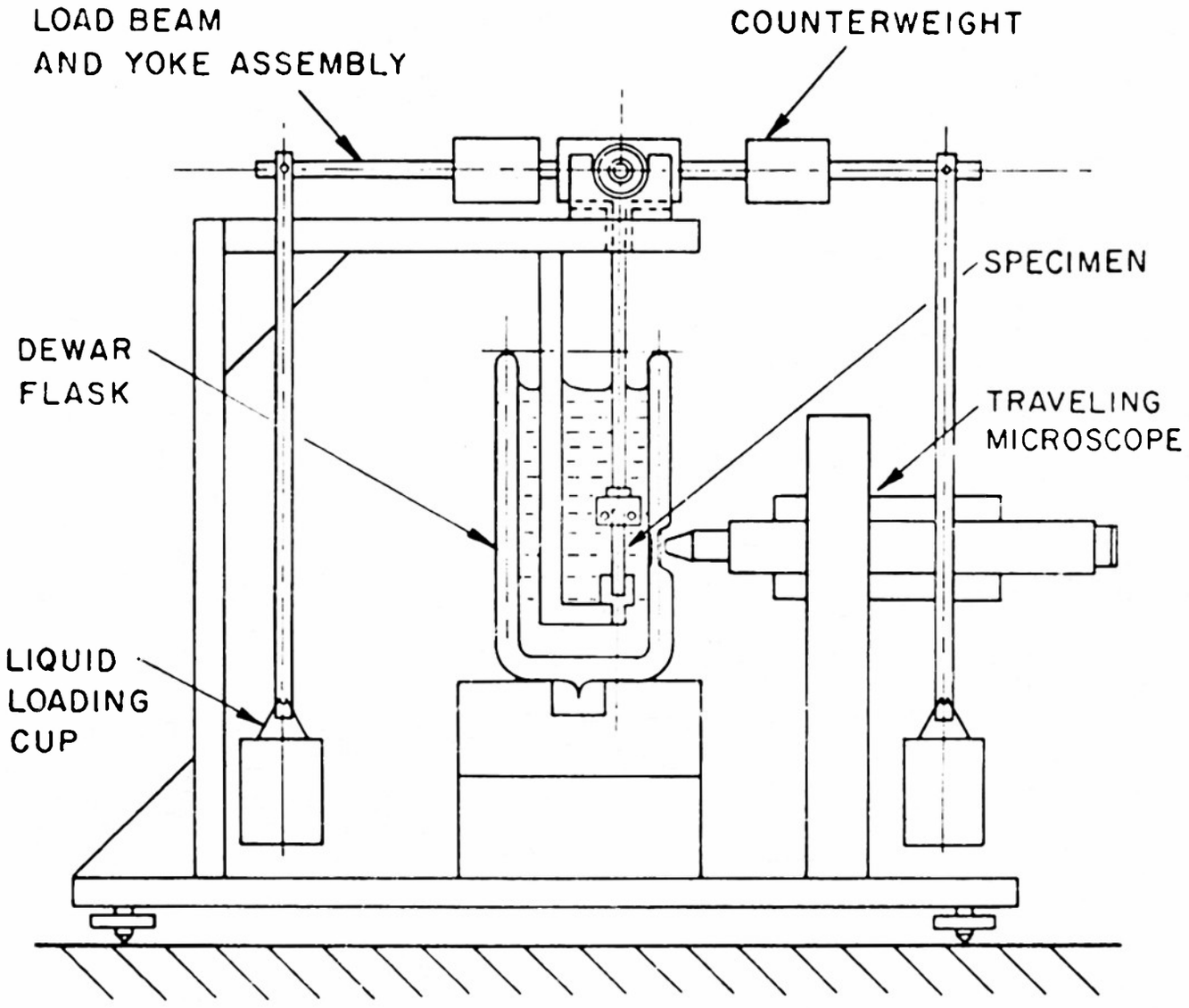


FIG. 3

SCHEMATIC DIAGRAM OF THE LOADING MECHANISM USED FOR BOUNDARY MOTION STUDIES

OBSERVATIONS AND DISCUSSION

A. Effect of Temperature and Stress on Boundary Motion

Small-angle boundaries used in this study consisted essentially of a planar array of edge dislocations with common Burgers vectors. These boundaries were made to move through the crystal by the application of a shear stress acting in the direction of the Burgers vector at temperatures varying from that of liquid nitrogen to near the melting point. The characteristics of this motion, however, vary markedly with temperature. In the higher temperature range, i.e. 300° to 400°C, the displacement rate of a boundary was observed to be constant under the action of a constant stress[4]. The rate was dependent upon the temperature, stress level, (Fig. 4) and the boundary angle (Fig. 5) such that the higher the temperature and stress or the lower the boundary angle, the greater the rate of boundary motion. At high temperatures it was not found possible to establish a lower limit of stress below which no motion would occur. The temperature dependence suggested that a thermal activation process was a major controlling feature. It was thus possible, as reported previously, to determine that an activation energy of approximately 21,500 cal/mole was involved in the motion of a boundary.

On the other hand, only a small amount of thermal assistance is available at room temperature for this process and essentially none at the temperature of liquid nitrogen. It was reasonable then to expect that boundaries would behave quite differently at these lower temperatures. In tests at room temperature, boundaries were observed to move in a manner remarkably different from that noted at elevated temperature. As the load was gradually increased, a value would be reached at which the boundary suddenly jumped to a new position at a velocity too rapid to be followed by the eye. The movement was frequently accompanied by a decrease in the angle of the boundary. The length of the jump was observed to be quite erratic. Jump distances as great as 0.5 mm were noted in some instances.

Motion of boundaries at liquid nitrogen temperature was different from either that at room or elevated temperatures. Neither creep under constant load as at

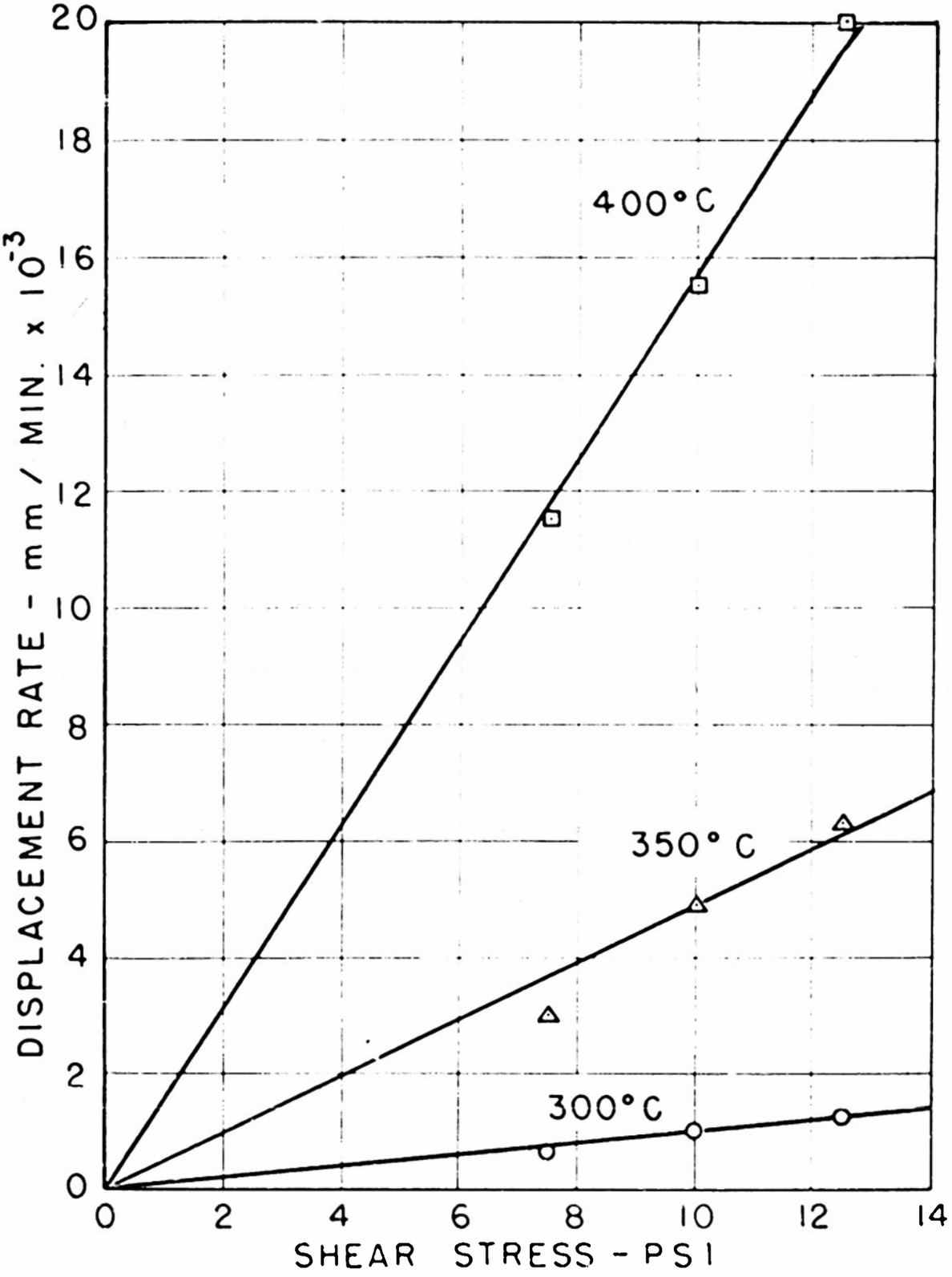


FIG. 4

DISPLACEMENT RATE OF A 2.0° EDGE DISLOCATION BOUNDARY AS A FUNCTION OF THE SHEAR STRESS

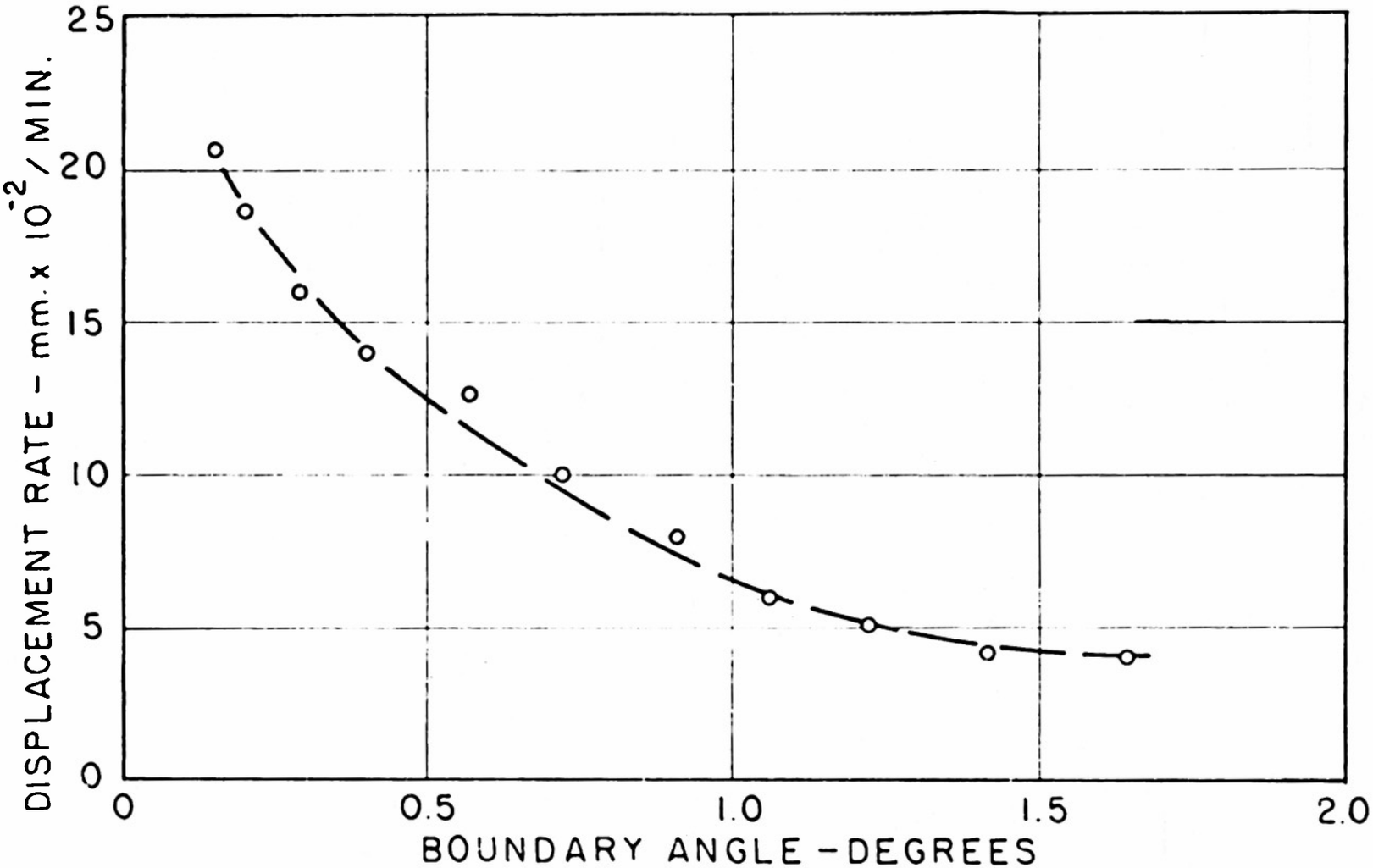


FIG. 5

DISPLACEMENT RATE OF EDGE DISLOCATION BOUNDARIES AS A FUNCTION OF THE BOUNDARY ANGLE UNDER THE ACTION OF A CONSTANT SHEAR STRESS OF 9.19 PSI AT 350° C.

high temperatures nor sudden jumps over appreciable distances as at room temperature were observed. Motion appeared to be steady when viewed at 50 diameters magnification but a continuously increasing stress was necessary to cause movement to continue. As at room temperature, the boundaries behaved somewhat erratically, changed character during motion, and occasionally refused to move even though the load was continuously increased.

B. Similarities Between Simple Shear Deformation and Boundary Motion

A comparison of the deformation of single crystals in simple shear with the motion of edge dislocation boundaries revealed some remarkable similarities. Single crystals of zinc tested at elevated temperatures exhibited steady-state creep characterized by a process having an activation energy of 20,000 cal/mole. This compared favorably with the activation energy for steady state boundary motion noted in the same temperature range.

Creep tests in simple shear on single crystals at room temperature showed that extension frequently occurred in a succession of sudden jumps. This phenomenon is apparently related to the jump-like behavior observed for boundaries at this temperature. Jerky motion of dislocation arrays lends strong experimental support to the concept of inter-locking of dislocations with immobile lattice imperfections.

The shear stress for motion of a dislocation array increases with decreasing temperature in a parallel fashion to the increase in critical stress for slip. The stress-strain curve for a single crystal tested in simple shear at liquid nitrogen temperature is given in Fig. 6. The stress-displacement curve of a 1° boundary obtained at the same temperature is shown in Fig. 7. In both cases a continuous increase in stress was necessary to maintain a constant displacement rate.

C. Changes in a Boundary as a Result of Motion

A dislocation array in a zinc crystal will be able to assume a low-energy configuration in a relatively short time at 400°C because of rapid diffusion.

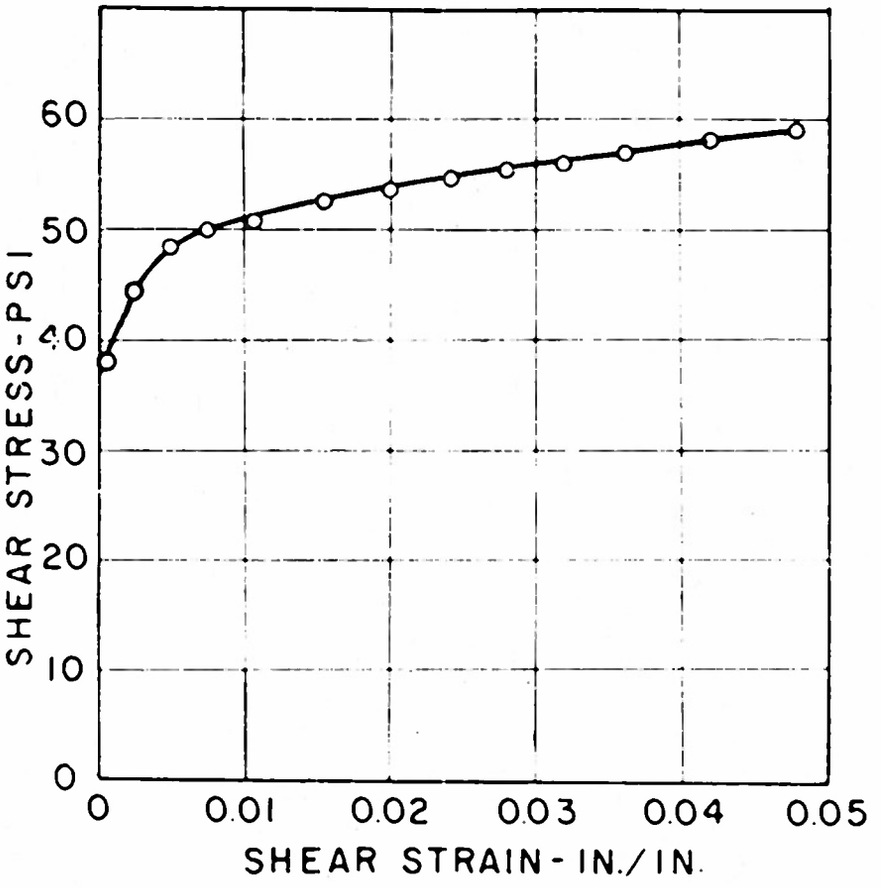


FIG. 6

SHEAR STRESS-STRAIN CURVE OF A Zn SINGLE CRYSTAL TESTED IN SIMPLE SHEAR AT -196° C.

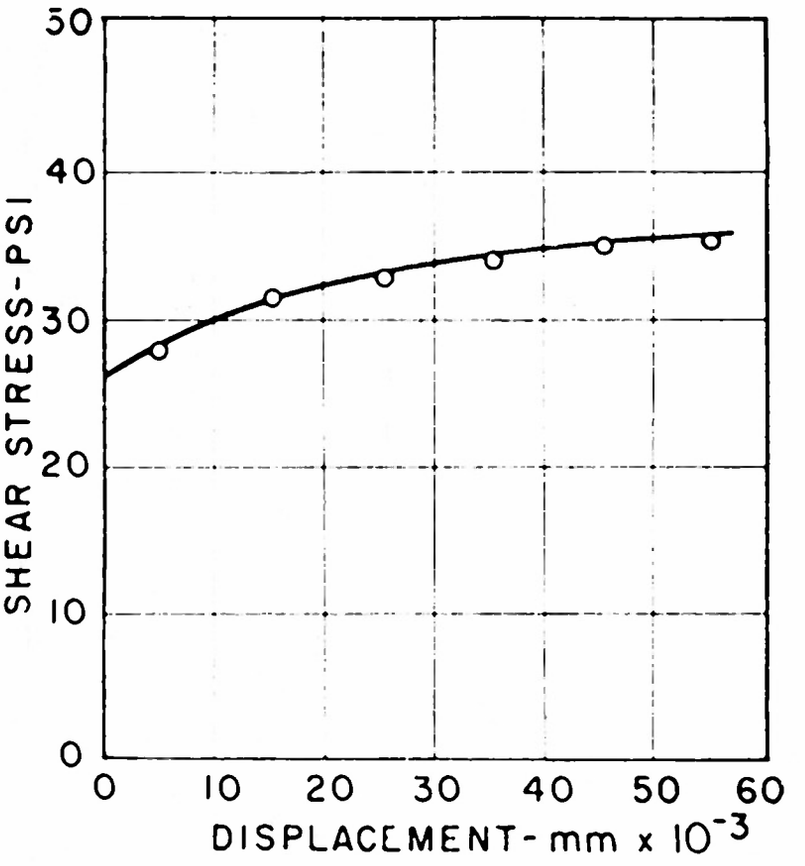


FIG. 7

STRESS-DISPLACEMENT CURVE OF A 1° ANGLE BOUNDARY PERPENDICULAR TO A SLIP DIRECTION AT -196° C.

Edge dislocations of like sign should distribute themselves into a fairly uniform planar array approximately at right angles to the slip vector of the dislocations. The degree of perfection which such a boundary array can attain is difficult to estimate, but it was assumed that after annealing the boundary angle was a reasonably accurate measure of the spacings of dislocations in the array.

When such a boundary is forced to move through the crystal by application of an external stress, the dislocations comprising the boundary presumably encounter obstacles; for example, immobile dislocation segments forming parts of a mosaic network. There is a possibility that during motion of the boundary, some of the dislocations in the array will interact strongly enough with immobile lattice imperfections so as to be left behind the moving array. In this event the boundary angle should decrease during motion. This was actually found to be the case. As shown by the goniographs of Fig. 8, when a boundary was moved about 0.25 mm at room temperature, the angle of the boundary decreased from 1.26° to 0.63°. Similar behavior was observed for motion at liquid nitrogen temperature. Despite the decrease in angular magnitude of the boundary, the stress necessary to move the boundary continued to increase. It is not yet possible to choose between numerous possible explanations for this continuous increase in stress. However, a few of the possibilities might be increase in the number of steps in the dislocation lines due to intersections with screw dislocation segments; back stresses attributable to trapped dislocations in the strained material through which the array has moved, increase in total density of dislocations in the boundary due to trapping of equal numbers of positive and negative dislocations generated in the bulk of the crystal during the test. The last possibility arises from the fact that the stress required for boundary movement is comparable with the yield stress of the crystal.

At high temperatures (300-400°C) the boundary angle did not change during motion. This fact is illustrated by the goniographs of Fig. 9. In addition, in this temperature range the boundary continued to move at a constant rate under

a. BEFORE MOTION OF THE BOUNDARY

b. AFTER MOTION OF THE BOUNDARY.

FIG. 8 PHOTOGONIOGRAPHS FROM A DISLOCATION BOUNDARY SHOWING THAT THE DECREASE IN THE BOUNDARY ANGLE FROM 1.26° TO 0.83° AFTER A DISPLACEMENT OF 0.25 MM AT 25 °C WAS ACCOMPANIED BY A DISSOCIATION OF THE BOUNDARY INTO A NUMBER OF SMALLER ANGLE BOUNDARIES.

a. BEFORE MOTION OF THE BOUNDARY.

b. AFTER MOTION OF THE BOUNDARY.

FIG. 9 PHOTOGONIOGRAPHS FROM A 0.89° DISLOCATION BOUNDARY SHOWING THAT THE CHARACTER OF THE BOUNDARY ESSENTIALLY WAS UNCHANGED AFTER A 0.50 MM DISPLACEMENT AT 375°C. (2 inches = 1°)

constant applied shear stress. This indicates that at temperatures where rapid diffusion is possible, any changes in the structure of the boundary and in the strained material through which it has passed can be continuously removed by a diffusion process. Trapped dislocations may be able to escape from immobile obstacles, and positive and negative dislocations captured by the boundary may annihilate each other by the mechanism of climb suggested by Mott[7]. Thus at higher temperatures the boundary can be pictured as maintaining a more or less constant structure during motion.

D. Boundaries as Barriers to Moving Dislocations

Frequently at liquid nitrogen temperatures or upon rapid loading at higher temperatures, a small angle boundary was observed to act as a very effective barrier against the motion of other dislocations. Fig. 10 shows a specimen which originally contained a 1° boundary at the center. Rapid loading at 375°C caused general slip to begin in the region of the specimen between the boundary and the place the load was applied. The bending resulting from this slip, however, was not general along the length of the specimen. As may be seen in the figure, bending occurred at the boundary near the center of the specimen, the angle of the boundary increased to 15°. This behavior clearly indicates the ability of a small angle boundary to act as a trap for moving dislocations. The specimen also contained another large angle boundary at the place the load was applied. Dislocations collected here because the shear stress fell rapidly from a high value to zero at this location.

E. Union of Dislocation Boundaries

When a number of small angle boundaries lie in proximity to one another in a zinc crystal, they may be made to join together by annealing to produce a single boundary having an angle equal to the sum of those of the individual boundaries. Fig. 11 shows the union of two boundaries resulting from a twenty-minute anneal at 400°C. It has also been observed that a "curved" region of the crystal

FIG. 10 MACROGRAPH SHOWING A 15° BOUNDARY DEVELOPED FROM A 1° BOUNDARY BY TRAPPING DISLOCATIONS GENERATED WITHIN THE CRYSTAL DURING RAPID LOADING AT 375°C. 4X.

a. BEFORE MOTION OF THE BOUNDARIES.

b. POSITIONS OF THE BOUNDARIES AFTER 5 MINUTES AT 400 °C.

c. UNION OF THE BOUNDARIES AFTER 20 MINUTES AT 400 °C.

FIG. 11 MICROGRAPHS SHOWING UNION OF BOUNDARIES OF LIKE SIGN AS A RESULT OF MOTION OF BOTH BOUNDARIES DURING AN ANNEAL AT 400°C. 200X.

near a boundary tends to straighten out and become flat during annealing. The goniographs of Fig. 12 show that the total angle remained constant. It is apparent that the union of boundaries reduces the strain energy of the crystal.

Dislocation boundaries of like sign can be made to unite by the application of a stress, even at low temperatures. Fig. 13 shows nine closely-spaced boundaries which were brought together at liquid nitrogen temperatures by a gradually increasing stress. The total angle of tilt was not changed by the unification. This stress-induced union of boundaries in the absence of thermal activation provides a mechanism for the building up of a substructure during deformation even at low temperatures.

It also was possible to unite boundaries of unlike sign both at high and low temperatures. The union of two such boundaries, shown in Fig. 14, was brought about by application of a constant stress at 375°C. The +0.49° boundary to the left and the -0.29° boundary to the right in Fig. 14a joined to form the +0.19° boundary shown in Fig. 14b. Although it has been observed that partially united boundaries of like sign may be separated again by a reversal of the applied stress, this has not been found to be true for partially joined boundaries of unlike sign. (Fig. 15) These experimental observations imply that the bringing-together of dislocations of opposite sign can result in their annihilation.

SUMMARY

Arrays of edge dislocations which produce low angle boundaries in zinc single crystals can be made to move by the application of an appropriate shear stress at temperatures from -196°C to 400°C. Continued motion at -196°C required the application of a steadily increasing stress. At room temperature the movement was discontinuous; during each advance, the boundary moved very rapidly through an appreciable volume of material. In contrast, at elevated temperature motion was steady and a thermally activated "creep" process seemed to predominate.

The deformation of zinc in simple shear showed characteristics very similar

a. INITIAL ARRAY.

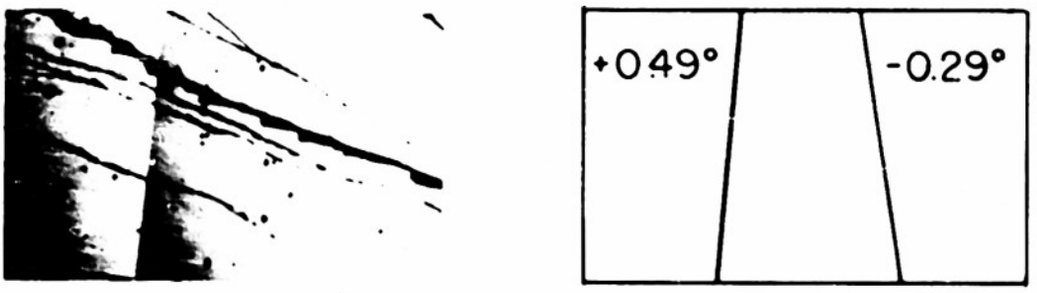
b. AFTER 5 MINUTES AT 375°C.

FIG. 12 PHOTOGONIOGRAPHS SHOWING THAN AN APPARENTLY CURVED REGION OF A CRYSTAL PECOMES PLANAR DURING AN ANNEAL AT 375°C. (2 INCHES = 1 DEGREE)

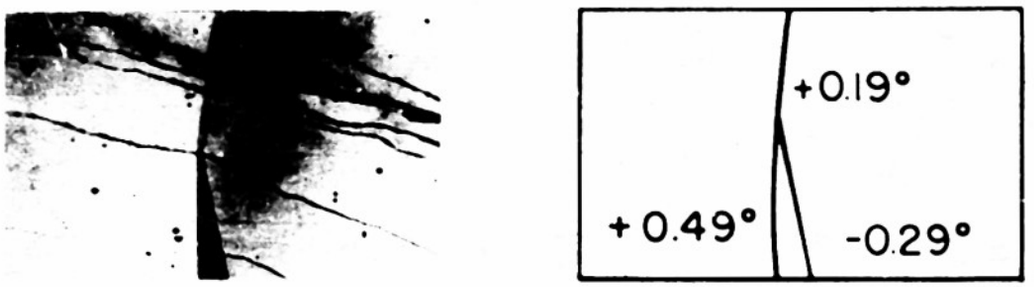
a. BEFORE MOTION OF THE BOUNDARIES.

b. AFTER MOTION OF THE BOUNDARIES.

FIG. 13 STRESS-INDUCED UNION OF BOUNDARIES OF LIKE SIGN IN A ZINC CRYSTAL AT -196°C. 400X.



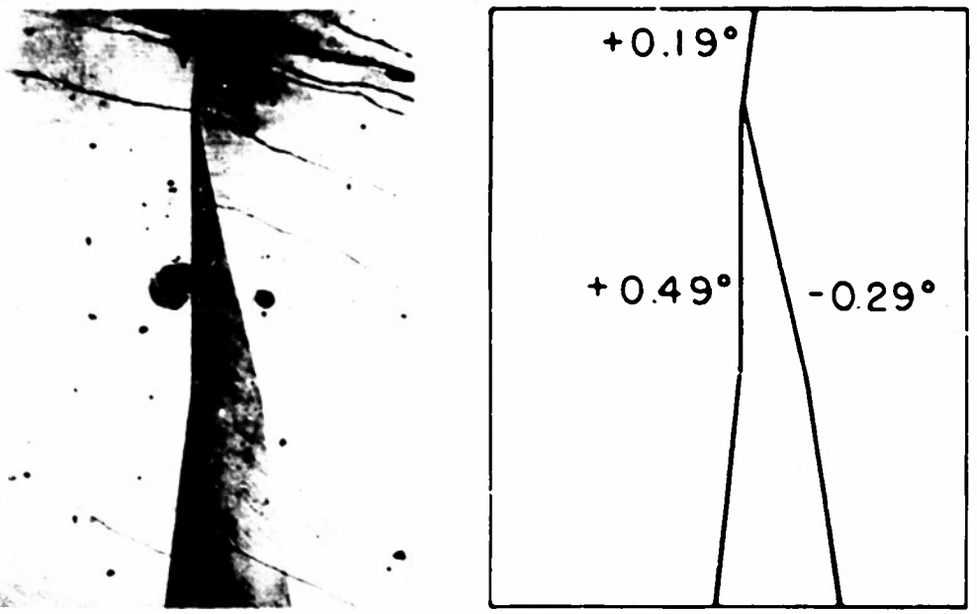
(a) BEFORE MOTION OF BOUNDARIES



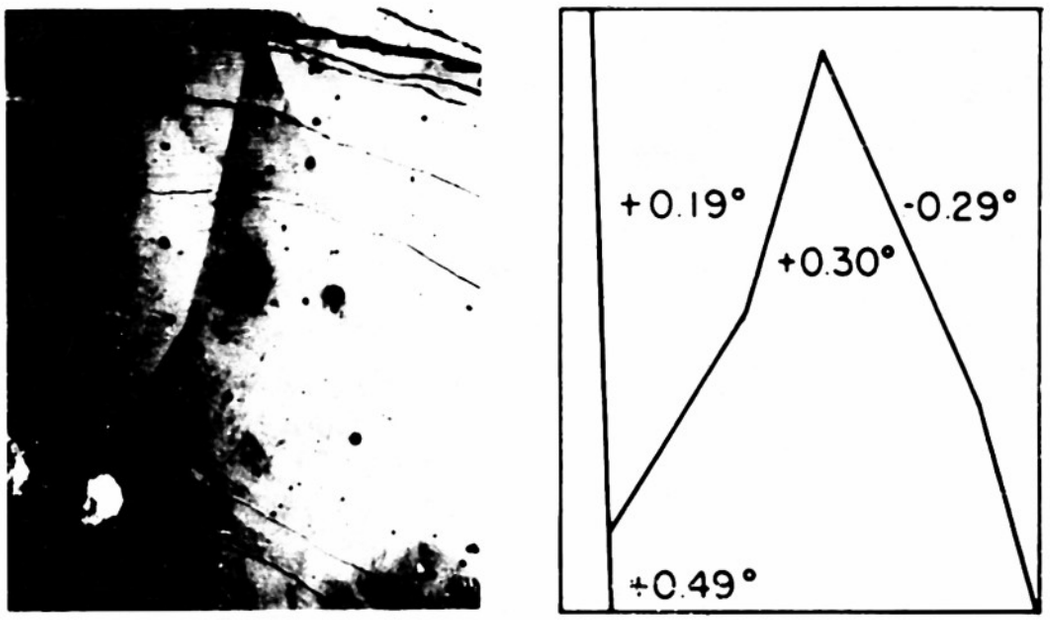
(b) AFTER MOTION OF BOUNDARIES

FIG. 14

STRESS INDUCED UNION OF BOUNDARIES OF UNLIKE SIGN AT 375° C. UNDER A CONSTANT SHEAR STRESS OF 10 PSI. 200 X



(a) BEFORE REVERSING THE DIRECTION OF APPLIED STRESS.
(SEE FIG. 14 b)



(b) AFTER REVERSING THE DIRECTION OF APPLIED STRESS.

FIG. 15

EFFECT OF REVERSING THE DIRECTION OF APPLIED STRESS AT 375°C ON THE MOTION OF THE BOUNDARIES SHOWN IN FIG. 14. THE +0.19° BOUNDARY FORMED PREVIOUSLY BY THE STRESS-INDUCED UNION OF THE -029° AND +0.49° BOUNDARIES DID NOT SEPARATE INTO THESE COMPONENT BOUNDARIES. THE CONTINUED MOTION OF THE +0.19° BOUNDARY CAUSED THE +0 49° BOUNDARY TO SPLIT INTO +0.19° AND +0.30° COMPONENTS 200 X

to those of moving boundaries at the same test temperatures. This implies that the propagation of dislocations rather than their generation may be the factor which determines the yield strength of zinc crystals.

During the motion of a boundary at elevated temperature, (300°-400°C), the boundary angle remained constant; at low temperatures the boundary changed in character both with regard to angle and regularity. Diffusion presumably assisted in overcoming barriers at the high temperatures so that the boundary could move as an integral unit. At low temperatures, however, portions of the boundary appeared to become trapped or were forced to assume more complex shapes through interaction with other structural defects.

Small angle boundaries of like sign in proximity to one another coalesced during short-time annealing treatments at 400°C to form a single boundary. "Curved" boundaries tended to become planar and perpendicular to the slip direction as a result of such treatment. Closely spaced boundaries of like sign were made to join at -196°C through the application of a stress, thus establishing a mechanism for the development of substructure in the absence of thermal motion of the boundary. Boundaries of unlike sign were also made to join by means of stress at both high and low temperatures. The angle of the boundary resulting from the union of boundaries of opposite sign at elevated temperatures is in accord with theoretical predictions that dislocations of opposite sign can be mutually destructive.

APPENDIX

Measurement of Small Angle Boundaries with an Optical Goniograph

For the purpose of determining the magnitude of the boundary angles in zinc, a goniograph employing optical reflection from the cleavage plane was used. The apparatus is shown schematically in Fig. 16. An intense light source emanating from a pinhole at "A" was focussed by means of a long focal length lens at "B" upon a plane ground glass or photographic film at "E" after approximately 90°

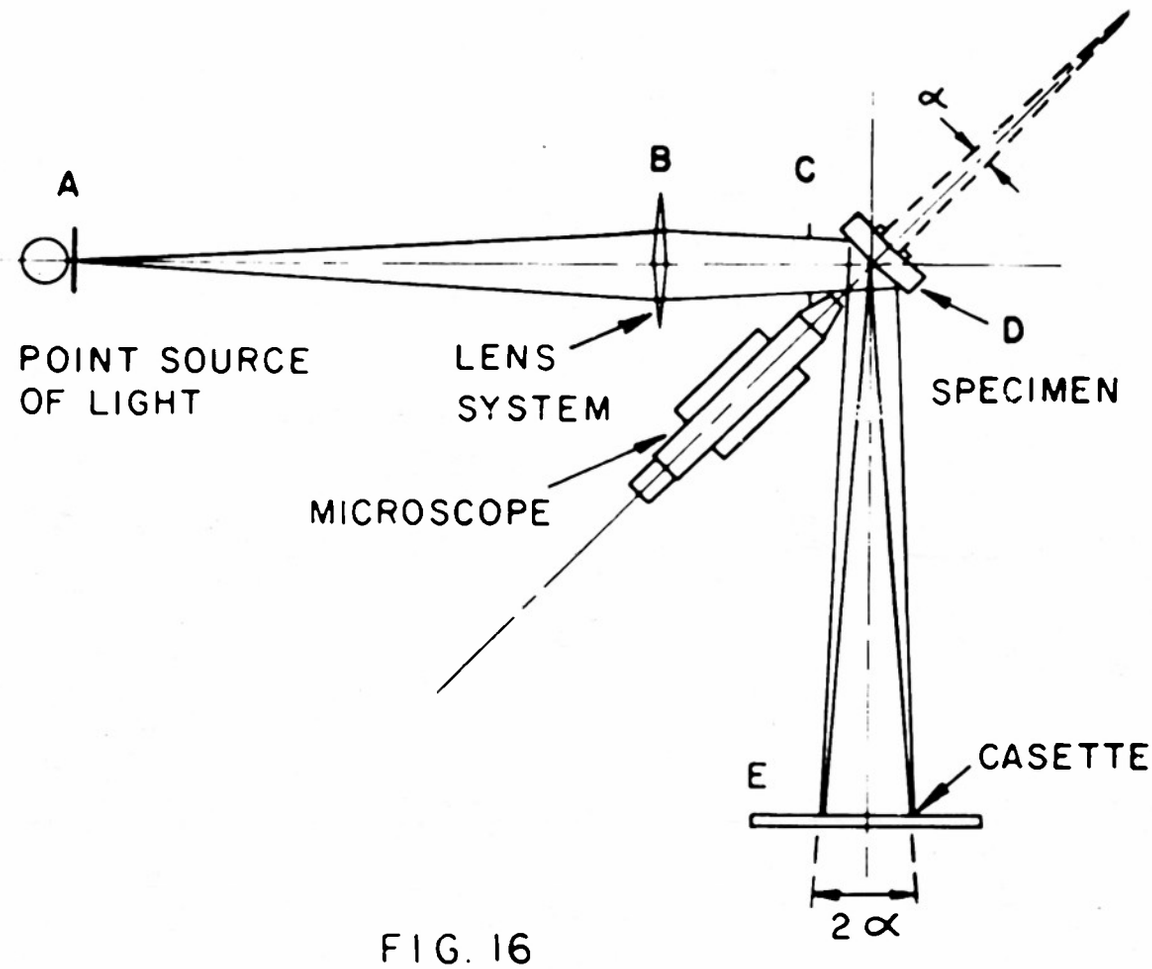


FIG. 16
SCHEMATIC DIAGRAM OF OPTICAL GONIOGRAPH

a. CIRCULAR APERTURE
2.54 mm. DIA. AT REFLECTOR

b. SLIT PERPENDICULAR
TO AXIS OF ROTATION
0.2 x 1.5 mm. AT REFLECTOR

FIG. 17

PHOTOGONIOGRAPHS FROM MIRROR TILTED THROUGH A SMALL ANGLE α. (ACTUAL SIZE)

reflection from a mirror surface, "D". The focussed spot at the position of the film thus had the shape of the pinhole source. The position of this focussed spot depended only upon the angle of tilt of the mirror and was otherwise independent of the position or extent of the mirror in space, provided that the reflecting plane passed through the point of intersection of the instruments optical axes.

Two reflecting planes differing in orientation by an angle of rotation about an axis perpendicular to the incident beam give rise to reflections differing by 2γ. Similar planes differing in orientation by an angle γ' about the incident axis yield reflections separated by the same angle γ.

In practice, a lens system with an effective focal length of 175 cm was employed in conjunction with a specimen to film distance of 72 cm. The focal spot was sufficiently large under these conditions to permit the film to be examined satisfactorily without magnification. A resolution of 0.008° was obtained with an optically flat mirror reflecting a 0.2° cone of light (2.54 mm diameter cross-section at mirror). It was considered desirable to be able to limit the reflecting region of the specimen such, for example, that variations in orientation, etc., occurring along a boundary might be evaluated. For this purpose a mount was supplied at "C", Fig. 4, in which slits or other apertures could be positioned. A slit 0.2 mm x 1.5 mm decreased the resolution considerably in a direction perpendicular to the slit, as shown in Fig. 17b, but essentially maintained the resolution in the plane of the slit. By positioning the slit and specimen such that the slit axis and the Burgers vector of edge dislocations comprising the boundary were both in the plane of incidence, optimum resolution in the desired direction was maintained. An 0.2 mm length of boundary was thus required for the angle measurements.

Alignment of a specimen in the goniograph was accomplished by means of a microscope whose axis bisected the optical axes of the instrument. This microscope was arranged to focus permanently on the axial intersection of the goniograph. The eye-piece cross-hairs established the axial plane and the position in

this plane of the intersection. A specimen to be studied was brought into position by means of a three coordinate translation device in conjunction with three leveling screws for rotational adjustment. The portion of the goniograph in the vicinity of the specimen is shown photographically in Fig. 18.

Reflections from zinc cleavage surfaces occasionally approached the resolution shown by the mirror but generally were considerably more complicated. In some instances this could be at least partly attributed to cleavage faults. In other cases, the influence of structural disconformities other than edge dislocations with a common Burgers vector appeared to be coming into play. An investigation of some of these effects is presently in progress.

FIG. 18 SPECIMEN STAGE OF OPTICAL GONIOGRAPH SHOWING CRYSTAL IN POSITION FOR BOUNDARY ANGLE MEASUREMENTS.

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